

EURACA #04

[A-16]

Aim: Preparation of EURACA FROM 7-Ac
by new route.

eq. to 50 gm 7-Ac

AGE T PREPARATION OF TFA

RAW MATERIALS

2 Euroyl chloride	4.23.0 ml
DMW	(350 + 15) ml
NASH (25% excess)	37.5 g
1:1 HCl	46.0 ml
1 DMW	175.0 ml
1 NaHCO ₃	20.4 g
1 EFOAC	100.0 ml
1:1 HCl	42.0 ml
1 EFOAC	250.0 ml

PROCEDURE

1. Charge DMW 350 ml & NASH at RT
Flush the funnel & 15 ml DMW
2. Stir at RT to get a clear soln
3. Add 2 Euroyl chloride in up. 45% at 20-25
then stir for 5'
4. Charge EFOAC 250 ml and then adjust
the pH to 1.0 - 0.9 by 1:1 HCl
at 20-25°C in 10-15'
5. Separate the layers
Give the O/L for HPLC analysis
6. To the O/L charge DMW (175 ml)
then adjust the pH to 0.0 - 0.2 by
NaHCO₃ at 20-25°C in 10-15'
7. Separate the layers
To the aq. layer charge EFOAC 100 ml
then adjust the pH to 1.0 - 0.9 by
1:1 HCl at 20-25°C
8. Separate the layers

Keep the org phase for 500 ml

Vol. of OL3 : 132.0 ml

HPLC Analysis Report

RT: 9.939 min

OL3 : 99.41

OL3 : 99.39

OL3 : 99.39

PREPARATION OF PURAC

RAW MATERIALS

2-ACA : 50.0 g

EEOAC : 200.0 ml

HOAC : 30.0 ml

BF3 : 68.5 g

TFA : 32.0 ml

DMF : 600.0 ml

SHS : 1.0 g

20% NH₄ OH

DMF : (50 + 150 + 50) ml } 1st part

EEOAC : (50 + 150 + 50) ml } 2nd part

PROCEDURE

Charge 2-ACA into the mix of

BF₃ purged EEOAC + HOAC at 15°C

Add the SHS for 5 l at 15°C

then add TFA soln. then raise

the temp to 30°C

Maintain the temp at 30°C

upto complete rxn.

After completion of rxn. transfer

the mass into the DMF (600 ml)

at 15°C then add SHS.

5 Adjust 160 - 10 - 10 - 2.5 3hr 18 - 20%

Net 201n of 20 - 25.6 in 25 - 30

6 201n for 201n 201n 201n

7 Filter 10 2 201n 5 201n 2 201n

DURABOND 201n 201n 201n 201n

7 ACA 2 TFA 201n 201n

45 7.92 19.43 20.51

75

1hr 15 2.55 113.67 21.49

1hr 45 1.10 11.92 26.44

2hr 15 0.45 11.09 18.52

Dry wt = 43.72g m/c - 2

Assay 93.9% (0.03)